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VOLUME 30 DECEMBER, 1952 NUMBER 12

THE USE OF A SIMPLE BAROGRAPH IN COSMIC RAY BALLOON FLIGHTS[1]

BY N. L. ALLEN[2] AND E. PICKUP

ABSTRACT

A self-recording barograph has been constructed and used successfully in determining the time–altitude records for free balloon flights carrying nuclear research emulsions to altitudes of about 100,000 ft.

INTRODUCTION

In order to obtain time–altitude records for free balloon flights carrying cosmic ray equipment to high altitudes, use has been made in the past (1,2)* of various types of self-recording barographs and other pressure recording devices, standard radiosonde and rawinsonde equipment, radar, and visual observations by means of theodolites. We have recently constructed a barograph which is simple, and robust in operation, and it was felt that a brief description of this instrument and its use in some balloon flights would be useful.

DESIGN AND CONSTRUCTION

The main features of construction are shown in Fig. 1, and Fig. 2 shows a photograph of the general arrangement. By means of a system of levers the expansion of an evacuated, thin metal bellows B is recorded on the drum D as altitude increases. When the bellows expands the end of the freely pivoted arm A_1 is lifted until it touches and raises the arm A_2, which is pivoted at P_2 and restrained by the spring S. Motion of A_2 is arranged so that it begins at an altitude of about 40,000 ft., and the movement is recorded on the smoked surface of the metal drum by means of the counterbalanced arm A_3, which serves to increase the amplitude of the vertical motion. The actual record is made by a pencil point on the end of A_3. Drum D is rotated, through a system of gear wheels, by the hour hand spindle of a Westclox "Baby Ben" clock.

An additional pivoted arm can be incorporated, if desired, similar to A_3, but to record a trace due to the movement of A_1 below 40,000 ft. For the present

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Contribution from the Department of Physics, National Research Council, Ottawa. Issued as N.R.C. No. 2838.
[2] *National Research Laboratories Postdoctorate Fellow.*
**This is not intended to be a complete bibliography.*

purposes this was not considered necessary, although provision for the arm was made as can be seen on the photograph in Fig. 2.

The bellows arrangement first used consisted of a nest of 10 pressure elements (as in Fig. 2) taken from standard radiosonde equipment, but that finally adopted was a 3 in. o.d., spring-loaded thin metal sylphon bellows (manufactured by Robertshaw-Fulton Controls Co., Knoxville), which was about the same over-all size as the bellows in the photograph.

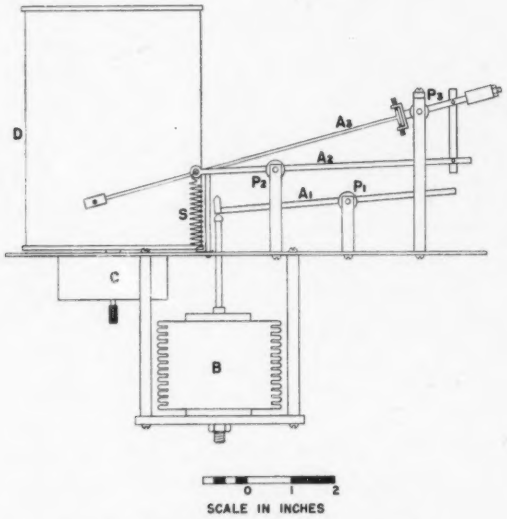

FIG. 1. Schematic drawing of barograph.

CALIBRATION AND FLIGHT TESTS

(*a*) *Calibration*

The apparatus was calibrated against pressure in a bell jar which could be evacuated, the pressure records being converted to altitude readings using standard tables. With the nest of radiosonde elements the deflection was approximately linear up to about 80,000 ft., but became very insensitive to changes in pressure for pressures corresponding to greater altitudes. Also, after expansion, and on contraction when the pressure was being increased, this bellows showed a pronounced hysteresis effect. The bellows finally used, inside which was mounted a compressed spring, was found to give a linear deflection with pressures decreasing to zero, and to show little hysteresis, the probable accuracy being $\pm$ 2000 ft. at 60,000-100,000 ft.

(*b*) *Flight Tests*

The barograph has been used in several balloon flights up to altitudes of 100,000 ft. It was mounted, together with nuclear emulsions for exposure to cosmic rays, inside an aluminum frame gondola enclosed by thin, white card-

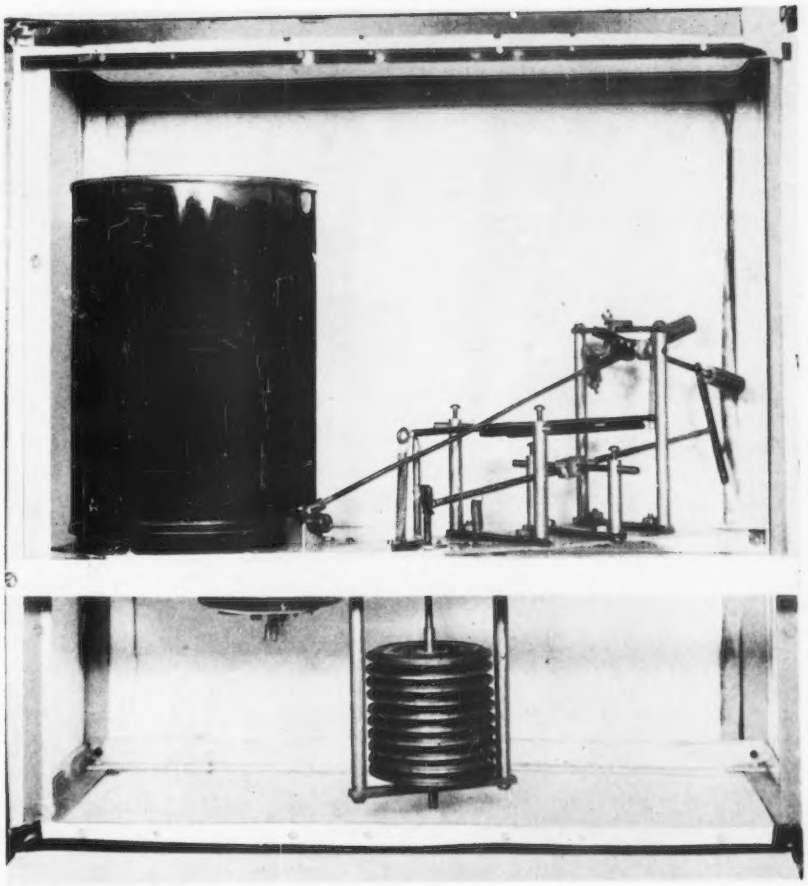

FIG. 2. Photograph of barograph mounted in gondola, after use in a balloon flight in Pennsylvania, July, 1951. The time-altitude trace on the smoked drum, beginning to the right at about 40,000 ft., indicates a maximum altitude of 100,000 ft. The base line is marked in half-hourly intervals.

board walls. The gondola was also surrounded by a double cover of thin cellophane, this arrangement being known to keep the temperature constant within a few degrees throughout the flights.

Fig. 3 shows a time–altitude record for a flight launched at Ottawa, the landfall being near Ogdensburg, New York. Points taken from the barograph record are in reasonably good agreement with those obtained from the bearings of two

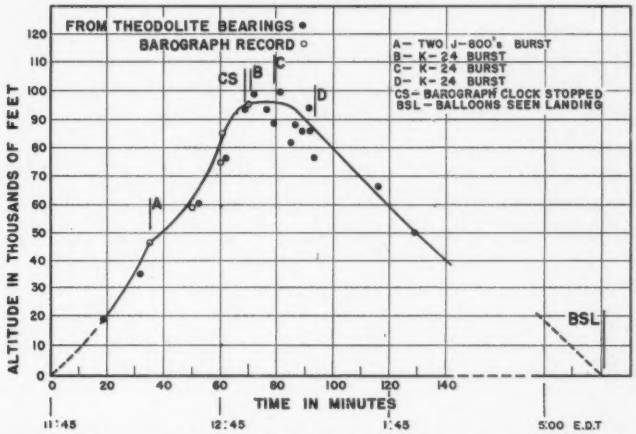

FIG. 3. Time–altitude record for balloon flight at Ottawa, June, 1952, showing heights from barograph and theodolite bearings. The barograph clock stopped at the maximum during this flight.

theodolites, indicating a maximum altitude of about 95,000 ft. The gondola was carried aloft on this flight by a cluster of six Dewey and Almy K24 balloons together with two J800 boosters to give a good rate of ascent. Balloons were seen to burst at various times by the theodolite observers, as indicated in Fig. 3. The accuracy of the theodolite results especially for the descending part of the record when the balloons drifted further away, was limited by a short base line between the two theodolites. Fig. 4 shows the barograph record for a flight with

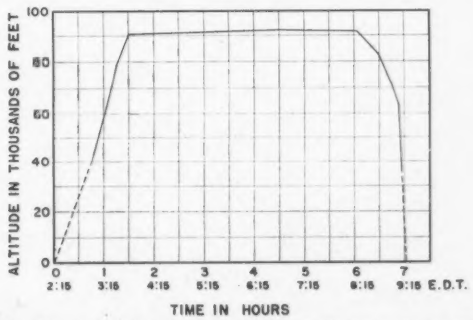

FIG. 4. Time–altitude record for balloon flight at Ottawa, June, 1952. The barograph record indicates level flight at 92,000 ft. for about four and one-half hours.

two J1400 and four K24 balloons, launched at Ottawa and recovered about 20 miles south at Vernon, Ont., indicating a level flight at 92,000 ft. for about four and one-half hours. No theodolite check was obtained for this flight owing to cloudy weather. An actual trace made on the smoked drum during a flight can be seen in Fig. 2, the instrument being carried on this occasion on a flight in Pennsylvania, organized by Dr. M. A. Pomerantz of the Bartol Research Foundation, using a cluster of about 20 J1400 balloons.

Sometimes a wide trace was obtained on the drum during the initial rise, rather than a clear line, this being suggestive of violent oscillations of the gondola during flight. This is probably connected with irregular motions within the balloon cluster in the winds of the upper atmosphere and with the bursting of balloons in the cluster, and will be corrected by stiffening the supports of the drum.

The authors wish to thank Messrs. H. A. MacDonald and D. Rushton for invaluable assistance in constructional work on the barograph.

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THE MEASUREMENT OF FIBER SWELLING BY THE LIQUID PERMEABILITY METHOD[1]

By M. Carroll[2] and S. G. Mason

ABSTRACT

The calculation of volume swelling of natural and synthetic fibers is described, based on measurement of the liquid permeability coefficient of carefully formed pads of fibers and the application of the Kozeny-Carman equation. Measurements carried out on solid filaments of nylon, acetate, and viscose rayon in a nonswelling liquid were in good accord with the specific volume values obtained by displacement in the same liquid. Measurements on the same fibers swollen in water showed only fair agreement with those calculated from moisture regain and from centrifuging measurements. The volumes of mixtures of a water-swollen cotton and sulphite pulp were found to be additive. It is concluded that the method is generally valid and potentially useful in studying swelling phenomena. The swelling of pulp fibers in different liquids and the swelling effect during beating are briefly considered.

INTRODUCTION

The phenomenon of swelling in natural and synthetic fibers is of fundamental importance in pulp and paper and textile technology. The physical and chemical properties of individual fibers and of fiber structures are highly dependent upon swelling produced by liquid immersion or vapor sorption, the most important swelling agent being water. The bonding together of fibers in paper, which determines most properties of paper, the chemical reactivity and accessibility of fibers, and the dyeing, shrinkage, and creasing properties of textiles are a few examples in which fiber swelling plays a governing role.

The direct measurement of swelling, i.e. the change of fiber volume, while simple in principle, is difficult in practice. Microscopic methods (14,15,18) are tedious, particularly with natural fibers where the geometry is irregular, and measurements must be limited to relatively small numbers, thus introducing serious sampling errors; with highly swollen and macerated fibers they are often inapplicable. Other methods such as those based on liquid displacement (4,19), bound liquid (3,20,21), and electrical conductivity (15) have serious theoretical and/or experimental limitations.

A rapid and convenient technique for the measurement of liquid swelling is that of Jayme (11) in which the mass of imbibed liquid is determined by centrifuging a suspension of fibers under standard conditions. Preston *et al.* (15,16) have extended the method and have proposed correction factors for liquid retained by capillarity in the interstices between fibers.

The investigation described here was undertaken to examine the applicability of the liquid permeability method. This work is an extension of an earlier

[1] *Manuscript received June 11, 1952.*
Contribution from Pulp and Paper Research Institute of Canada and Chemistry Department, McGill University, Montreal, Que.
[2] *Holder of a National Research Council Fellowship. Present address: Celanese Corporation of America, Summit, N.J.*

study, the primary purpose of which was to measure specific surface of fibers (17). Measurements on beaten wood pulp fibers reported at that time indicated imbibition of as much as 500% of water by the dry fibers. This is considerably higher than that found for similar pulps by the centrifuging method, and the permeability values have been questioned by Gallay (7). The permeability method has been assumed valid in interpreting the electrokinetic behavior of fibers (8). For these reasons, and because of the need for alternative means of measuring fiber swelling, it was considered desirable to study the method further.

The principles and the experimental requirements of the method have been described in detail elsewhere (17), but the essential idea is as follows:

The rate of viscous flow of a fluid through a uniform bed of fibers, expressed as the permeability coefficient K, may be related to the internal pore structure by the rectilinear form of the Kozeny-Carman equation:

$$(Kc^2)^{1/3} = (1 - \alpha c)/(k\sigma^2)^{1/3} \tag{1}$$

using the symbols defined under *Nomenclature*. If Eq. (1) applies, a plot of $(Kc^2)^{1/3}$ against concentration (c) of fiber in the bed yields a straight line whose

NOMENCLATURE

A = cross sectional area of cylindrical pad of fibers.
c = mass (dry basis) of fibers per unit volume of pad.
K = permeability coefficient = $\frac{Q\eta L}{A p}$
k = shape-orientation factor of Kozeny-Carman equation (6).
L = length of pad.
M = mass of pad (dry basis).
p = pressure difference across pad causing flow.
Q = volume rate of flow of liquid.
α = effective volume of unit mass (dry basis) of swollen fibers.
αc = "hydrodynamic" solid fraction.
η = viscosity of liquid.
σ = "hydrodynamic" surface area per unit mass (dry basis) of fibers.

intercepts are $1/k\sigma^2$ and $1/\alpha$. The quantity α, identified here as the effective volume of the swollen fibers, is the volume per unit mass of dry fiber which excludes flow of the liquid. Strictly speaking it should be referred to as the "hydrodynamic volume" (13).

In the measurements described below, it is shown that α determined by the permeability method for a variety of fibers shows reasonable agreement with the true fiber volume for cases where a direct comparison can be made. Evidence is presented to show that the discrepancy between results for wood pulps by the permeability and centrifuging methods is a result of the compressive stress applied to the pulp during centrifuging, which results in a partial de-swelling.

EXPERIMENTAL PART

Permeameters

Two permeability cells of similar design were used. Each consisted of a vertical cylinder in which the pad was confined between two 60-mesh wire screens, one attached to a perforated brass support and the other to a perforated brass piston. Outlets from the cylinder, above and below the pad, were con-

nected to a differential manometer and to the inlet and outlet flow lines containing constant level overflow devices.

For highly swollen fibers ($\alpha > 1.5$ cc. per gm.) where the pads were easily compressed, a glass cylinder was used; the pad concentration was varied by applying weights on the piston. For the more rigid fibers an all-brass cell (Fig. 1)

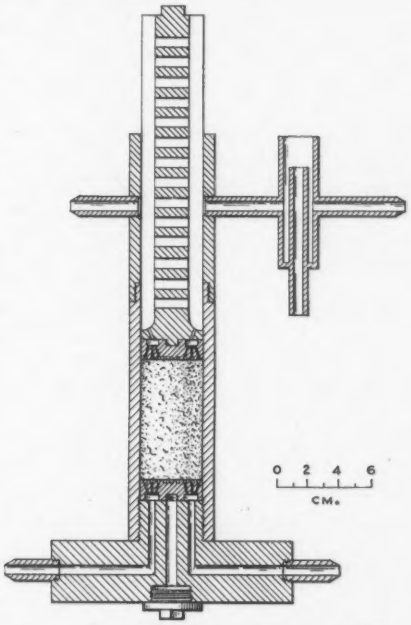

FIG. 1. The high compression permeability cell.

of rugged construction, fitted with a slotted and drilled solid brass piston, was used. In this cell, loads (up to 1.5 tons) could be applied to the piston to compress the pad by using an inverted scissorbill screwjack.

Pad Formation

To obtain reproducible measurements, previous experience (8, 17) showed that the pads must be homogeneous and free from entrapped air.

Fines and long fibers were removed by wet screening and the fraction retained between 20 and 150 mesh was used. When filament yarn or long staple was used, the dry fibers were chopped in a Wiley mill before fractionating. The range of fiber lengths provided by fractionating between these limits was found suitable for forming uniform pads. Fibers retained on the 20 mesh screen cause excessive particle aggregation during the forming stage and tend to give lumpy pads. Fines, particularly with wood pulps, make the pad slow draining and the procedure, both of formation and measurement, is tedious. In addition axial concentration gradients are often set up during pad compression.

A pad was formed in the cylinder of the permeameter by a combined process of filtration and sedimentation from a dilute deaerated suspension. This was poured into a large funnel fitted to the top of the cylinder. A motor-driven stirrer was placed in the funnel to control sedimentation and to prevent clogging of the funnel neck. Flocculation of the fibers in the cylinder was controlled at a minimum practicable level by adjusting the suspension concentration and by agitating the suspension in the cylinder by means of a four pronged hand stirrer extending through the neck of the funnel into the cylinder body.

Measurement

The freshly formed pad was compressed to an initial solid fraction of approximately 0.2. With the piston locked in this position, a flow rate measurement was made after adjusting the constant level devices to obtain a flow rate not greater than 25 ml. per min. The temperature of the effluent liquid, the pressure, and the pad length (measured by means of a cathetometer) were all recorded. The pad was then compressed in steps and the measurements repeated at each stage.

From this data K was determined for each value of L. Since $c = \dfrac{M}{AL}$, where M is the dry mass of the pad, Eq. (1) can be expressed in the alternative form

$$(K/L^2)^{1/3} = (A^2/k^2M^2)^{1/3}(1 - \alpha M/AL). \tag{2}$$

As a matter of convenience the data were usually plotted as $(K/L^2)^{1/3}$ vs. $1/L$ and the intercepts of the best fit straight line were determined analytically. M was subsequently determined by oven-drying to constant weight at 105°C., and values of α and ko^2 calculated from the intercepts. In evaluating σ, Fowler and Hertel's (6) value of $k = 5.55$ was used.

Reproducibility

The many hundreds of measurements carried out in this and earlier studies (17) show that the rectilinear form of the Kozeny relation is obeyed over a limited range of pad concentration. The lower limit of αc, imposed by theoretical considerations (5), was found to be around 0.2 and is illustrated in the plot for cotton in Fig. 2. The upper limit of c is governed either by the maximum load which can be applied in compressing the pad, or in the case of highly swollen natural fibers containing a lumen void, by the pressure limit beyond which deformation of the fiber by collapse of the lumen, or deswelling of the fiber wall, occurs to cause a decrease in α and a resulting deviation from linearity. An example of nonlinear behavior at high pad concentrations is shown in Fig. 3. Generally speaking, this occurred with water-swollen cotton and wood pulp fibers when $\alpha c > 0.5$.

The main experimental difficulty was the decay in permeability at fixed pad concentration which had previously been observed (17). Systematic experiments, details of which we omit, showed that (1) it was most serious with highly-swollen and/or very flexible fibers, (2) it was not due to felting at the pad supports or to the movement of fines, as we had been earlier led to suppose, and (3) decay was accelerated by liquid flow. The observations suggested that the

decrease in K was due to a relaxation of internal stresses in the compressed pads, causing a movement of the fibers which in turn caused k to increase progressively.

When decay occurred, it was minimized by measuring the flow rate as quickly as possible, immediately releasing the stress on the pad, and momentarily reversing the direction of liquid flow, before proceeding to the next measurement at a higher pad concentration.

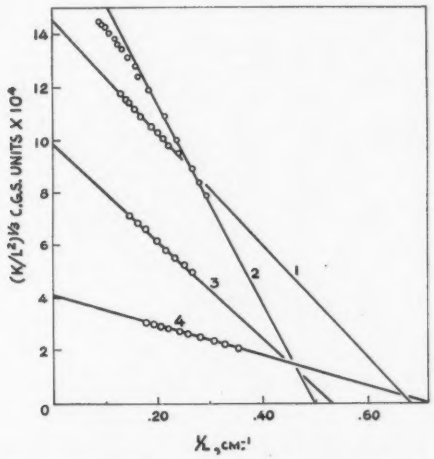

FIG. 2. Permeability plots in the form of Eq. (2) for nylon (Curve 1), cotton (Curve 2), bleached sulphite pulp (Curve 3), and hitenacity rayon (Curve 4) using kerosene.

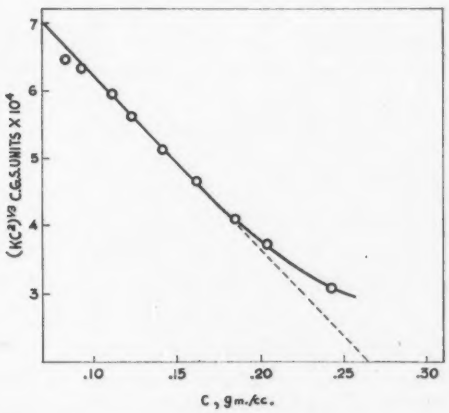

FIG. 3. Permeability plot in the form of Eq. (1) for bleached sulphite pulp and water. Note the deviation from linearity at the higher pad concentrations.

The method presupposes that at a fixed pad concentration $\frac{Q\eta}{p}$ is independent of p. An interesting exception to this was found in the case of a beaten (highly swollen) soda pulp. Here a substantial increase in $\frac{Q\eta}{p}$ with p was found in a pad of fixed length. This is illustrated for $1\frac{1}{2}$ pressure cycles in Fig. 4. Because of the prolonged period of the measurements appreciable decay occurred;

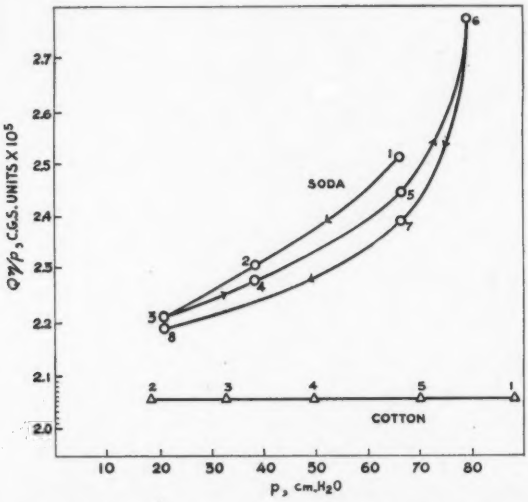

FIG. 4. Increase of $Q\eta/p$ with p for a pad of beaten soda pulp. The points are numbered in the order in which the measurements were made. Similar values for cotton, showing normal behavior, are included for comparison.

however, it is clear that there was a *recoverable* increase in K as p increased. The change in K was sufficiently great to invalidate the method for this material.

Table I shows a series of results obtained for a series of fibers in both swelling and nonswelling media, and shows that when the precautions described above are observed the reproducibility of α is good. Of the factors governing the reproducibility, the most important, apart from uniform pads, are (1) permeability decay and (2) the range of pad concentration over which a linear plot of the data can be made. The latter determines the accuracy of extrapolation as is evident from Fig. 2 where the origins of both axes are shown.

Significance of α

In order to establish whether α represents the true fiber volume, measurements were carried out on a number of fibers in a nonswelling medium; for convenience, kerosene was used. The values found (Table II) for the solid fibers compare favorably with those determined pycnometrically. With sulphite pulp, the specific volume is about 25% lower than α. Graff *et al.* (9) have shown from

TABLE I

REPRODUCIBILITY OF α FOR VARIOUS FIBERS

Fiber	Liquid	No. of determinations	α, cc./gm.	M.D., %
Undried bleached sulphite pulp	Water	7	3.34	1.2
Dried bleached sulphite pulp	Water	2	1.31	1.0
Nylon	Water	4	0.98	1.0
Acetate	Kerosene	2	0.77	0
	Water	2	0.90	0
Hitenacity rayon	Kerosene	3	0.64	0.5
	Water	2	1.36	0.8

TABLE II

FIBER VOLUMES IN KEROSENE

Fiber	α, cc./gm.	Specific volume, cc./gm.
Nylon	0.88	0.88
Acetate	0.77	0.77
Hitenacity rayon	0.64	0.65
Bleached sulphite pulp	0.84	(0.64)*

* Estimated.

microscopic studies that dryed softwood fibers have uncollapsed lumen volumes of the order of 20% of the total fiber volume, so that the difference observed here is not unreasonable.

Measurements were also made on the solid fibers swollen in water. Here the true swollen volumes were estimated by rough measurements of the moisture (vapor) regain at saturation humidity. The fiber volume was calculated on the assumption that the effective volume of the sorbed water is that of the condensed liquid and that its volume can be added to that of the unswollen fiber to give a sufficiently accurate value of the swollen volume as has been shown by Hermans (10). Nylon and acetate show good agreement (Table III) whereas the viscose rayons (which have a high moisture regain) yield α's higher than the calculated values. This discrepancy can be attributed to the difficulty of determining reliable values of regain in the saturation region; it is conceivable that the moisture values are too low.

As a further check, Preston and Nimkar's (15) modification of Jayme's (11) centrifugal method was used, the somewhat arbitrary corrections being made for capillary bound water from the data of Preston and Nimkar for similar fibers. The agreement with α is fair (Table III), and in the case of the hitenacity rayon somewhat better than with the values based on moisture regain.

A similar comparison was made with a beaten sulphite pulp, although here corrections for capillary bound water could not be made; the centrifuge values

TABLE III

FIBER VOLUMES IN WATER

Fiber	α, cc./gm.	Swollen volume cc./gm. dry basis	
		From moisture regain	From centrifugally retained water
Nylon	0.98	0.98	—
Acetate	0.90	0.93	0.95
Hitenacity rayon	1.37	1.07	1.35
Standard rayon	1.20	1.01	1.38

should therefore be too high. The centrifuge and permeability methods yielded values of 3.0 and 4.0 cc. per gm. respectively. It is significant, however, that the final pad concentrations in the centrifuged material was 0.26 cc. per gm. The permeability plot for this type of pulp was found (cf. Fig. 3) to deviate from linearity at $c > 0.16$ cc./gm., an effect which we have interpreted as being caused by deswelling of the fibers by the compressive stress applied to the pad. In the light of this evidence, we believe that the discrepancy between the two methods is due mainly to the compressive stress generated by the centrifugal field.

Since no reliable independent methods of checking α for water swollen wood pulp fibers were available, it was considered worthwhile to carry out a series of measurements using various mixtures of cotton ($\alpha = 0.96$ cc. per gm.) and beaten sulphite pulp ($\alpha = 4.00$ cc. per gm.). The results are shown in Fig. 5. The linearity of α vs. composition shows that it is additive.

The results of these experiments were taken as evidence that the permeability method provides a satisfactory method of measuring the free swollen volume up to values of $\alpha = 4$, provided that suitable experimental conditions are chosen.

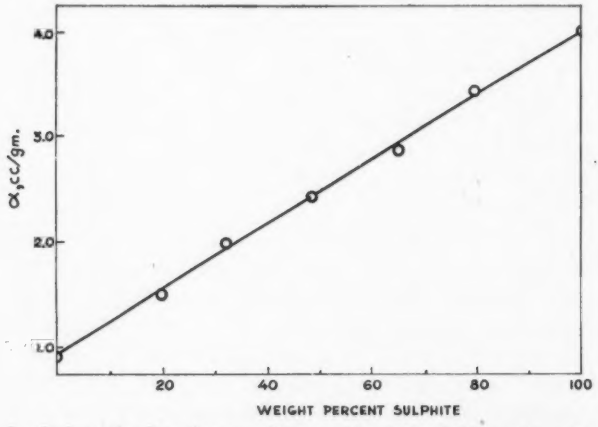

FIG. 5. Values of α for mixtures of beaten bleached sulphite pulp and cotton.

APPLICATION

We wish now to consider briefly several applications of the permeability method to the swelling of wood pulps.

1. *Effect of Fiber Length*

In the Experimental Part, it was mentioned that removal of long fibers and fines facilitated the formation of uniform pads. Experiments were carried out to determine the effect of removing the objectionable fractions. To obtain a reasonable length distribution, a sample of air-dry, bleached sulphite pulp was chopped in a Wiley mill. This was then wet classified in a Bauer-McNett classifier, and the fractions allowed to soak in water for 10 days to come to swelling equilibrium. A second sample of the chopped pulp was beaten to 380 Canadian Standard Freeness in a Valley laboratory beater prior to wet classification.

These measurements were made in the early stages of the investigation, before a number of improvements were made, and the accuracy of the results was not high. The α and σ values for the various fractions are shown in Table IV. The values marked by asterisks are uncertain as these fractions did not form homogeneous pads. It is seen, however, that the α of any one fraction is fairly representative of the whole. The σ value of the whole pulp, especially in the case of the beaten pulps, is due largely to the fines. We have previously shown (17)

TABLE IV

α AND σ VALUES FOR VARIOUS FIBER LENGTH FRACTIONS

Fraction	Unbeaten pulp		Beaten pulp	
	α, cc./gm.	σ, cm.^{2}/gm.	α, cc./gm.	σ, cm.^{2}/gm.
0–20 mesh	1.8*	7300*	2.2*	14000*
20–35	1.9	8600	3.3	17000
35–65	1.9	9000	3.4	15000
65–150	1.9	8800	3.4	19000
Whole pulp	1.85	16400	3.2*	44000*

* See text.

that the freeness test provides a rapid empirical measure of σ. Clark (2) has criticized the freeness test for evaluating pulp properties on the ground that the value is determined largely by the fines. To the extent that the freeness test is a measure of surface area, Clark's argument is supported by the data given here.

2. *Swelling and Paper Strength*

Robertson and Mason (17) found evidence of a relationship between the increase of swelling and the development of paper strength in a pulp during beating. Their permeability measurements were made on whole pulps, and in consequence of the experimental difficulties described earlier, the accuracy of the swelling measurements was low. The experiment was repeated, the experimental conditions being modified to improve the accuracy.

A sample of bleached sulphite pulp sufficient for six beater runs was chopped in a Wiley mill to eliminate the bulk of the long-fiber fraction. Aliquots of this material were beaten under TAPPI standard conditions in a Valley laboratory beater (capacity 350 gm.) for various times up to 55 min. The entire contents of the beater at each beating time were classified in a Bauer-McNett classifier to obtain the 35 to 65 mesh fraction. Test handsheets were formed from this fraction and a sample was also reserved for permeability measurement.

Fig. 6 shows the tensile, burst, and tear strength, and α plotted on a beating time scale.

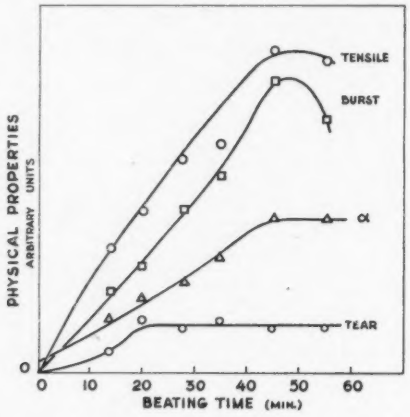

FIG. 6. Parallel increase in fiber swelling in the wet state and of paper strength during beating.

Several significant points are indicated by the data.

1. The swelling reached a maximum at approximately 45 min. beating time. Microscopic examination of the 35 to 65 mesh fractions at the various times of beating showed very little evidence of visible fibrillation (other than at isolated points where the fiber had been crushed or cut in the beater) until the maximum strength was attained.

2. The tensile and burst strength both reached a maximum at the same time as α reached a maximum. Since the test sheets all contained a roughly similar distribution of fiber lengths, a general relationship between these two strength properties and α is indicated. The decline in tensile and burst strength after reaching a maximum observed with whole (i.e. unfractionnated) pulps is generally attributed to the shortening of the fibers by the cutting action of the beater. The fact that the same general behavior was shown here casts doubt on this explanation.

3. The tear strength rose quickly to a maximum value and then remained constant. With whole pulps, the tear generally rises rapidly and then declines. The result observed here is evidence of the dependence of tear mainly on fiber length.

The experimental data presented here are limited, and no general conclusions may be drawn. The results, however, confirm previous observations and are in accord with views expressed by Gallay (7) of the importance of swelling as a measure of the extent of beating.

3. Swelling in Various Media

Swelling measurements were made on oven dried samples of bleached sulphite pulp using liquids of various swelling powers. Values of α and the percentage increase in volume over the kerosene value are given in Table V. A plot of the

TABLE V

SWELLING OF BLEACHED SULPHITE PULP IN VARIOUS MEDIA

Liquid	α, cc./gm.	Per cent increase in volume
Kerosene	0.83	0
Amyl alcohol	0.92	9
Propyl alcohol, 95%	1.02	19
Ethyl alcohol, 95%	1.08	25
Methanol	1.12	29
Water	1.36	44

percentage increase in volume vs. dielectric constant is shown in Fig. 7. Data taken from the work of Kress and Bialkowsky (12), obtained from measurements of the change in bulk volume of compressed pads of fibers, are plotted for comparison and, although determined by an entirely different method and on a different sample, they show parallel behavior. The choice of dielectric constant as abscissa is largely a matter of convenience, although it is to be expected that the swelling of a partially hydrogen-bonded material like cellulose will be related to the polarity of the swelling medium.

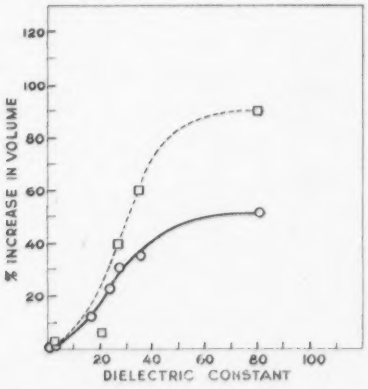

FIG. 7. Swelling volume of bleached sulphite pulp fibers in various liquid media (cf. Table V). The broken line shows values reported by Kress and Bialkowsky using a different experimental method.

DISCUSSION

With solid unswollen filaments, the permeability method yields fiber volumes in good agreement with directly determined values. When swollen in water, the agreement with values calculated from vapor sorption and from centrifugally retained liquid is only fair; because of known limitations of the last two methods, we are of the opinion that the permeability measurements give the correct values.

With water-swollen wood pulps, where α is large, the difference between the permeability and centrifuging values for sulphite pulp has been partially accounted for by the compressive stresses acting to de-swell the fiber; in the absence of direct microscopic measurements, however, we cannot state unequivocally that the permeability measurement gives the correct absolute value. There is no doubt, however, that it indicates changes in fiber volume which are in the expected order.

A complete permeability run takes from two to four hours, and is thus less rapid than the centrifuging method. On the other hand, it has distinct advantages over the microscopic method; it is significant that the latter method has rarely been used in systematic experimental studies of fiber swelling phenomena.

The nonlinear flow observed with the highly swollen soda pulp (Fig. 4), rendering the permeability method inapplicable, is of interest. A possible explanation of this effect is that pressure gradients set up by the flowing liquid cause a recoverable distortion of the fibers in such a way as to decrease resistance as the flow rate increases. Adamson (1) has reported a nonlinear effect in pads of wool in the opposite direction to that found here, and has attributed it to a change in the pattern of streamline flow with increasing flow rate i.e. as an effect comparable to the onset of turbulence. Since the experimental details are not given, however, it is not possible to relate Adamson's observations to those given here. The phenomenon reported here appears to be of sufficient interest in connection with fluid flow through porous materials to warrant further investigation.

ACKNOWLEDGMENT

The authors wish to acknowledge the assistance of Mr. R. H. Marchessault in some of the permeability measurements.

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BARITE-AGGREGATE CONCRETE FOR GAMMA-RAY SHIELDING[1]

By K. A. Shelstad, V. E. Vaughan, and E. L. Cameron

ABSTRACT

Data concerning the preparation, physical properties, and gamma-ray attenuation of concretes prepared using barite as aggregate are reported. Concrete of density from 190 to 200 lb. per cu. ft. is readily obtainable. Mechanical strength is limited by the friable nature of the aggregate rather than by the strength of the barite–cement bond, as indicated by visual observation of fractured specimens. Subject to this limitation, there are no inherent difficulties in using barite as aggregate. Cement–aggregate bond is good and water–cement ratio is normal. Deterioration with time was not studied.

Attenuation of gamma rays (1.1 to 1.3 Mev.) under narrow-beam conditions can be predicted from a knowledge of the concrete density and using a mass absorption coefficient of 3.9 sq. in. per lb.

INTRODUCTION

The potential danger of atom bomb attacks raises the question of protection against both high energy radiation and the blast effect of bombing. The essential need is a structure with effective protection against these hazards and at the same time requiring a minimum of scarce structural materials.

The Defence Research Board of Canada, during 1950, sponsored an investigation into the use of barite (barium sulphate rock) as a possible high density concrete aggregate. This paper reports on the measurements made of the mechanical and working properties of barite-aggregate concrete. Included also are the results of experiments on the attenuation of gamma rays from cobalt-60 under narrow-beam conditions.

Inclusion of barium increases the shielding properties of concrete because of the high material density obtained. The reduction of secondary radiation from the barrier may be important also because of the large photoelectric cross section of barium at energies below 0.4 Mev. Data concerning this latter point were not obtained during this investigation.

The processes by which gamma rays are attenuated have been discussed elsewhere (1).

EXPERIMENTAL

Concrete Specimens

The barite used was a relatively low grade material possessing little commercial value. The ore shipment, weighing approximately two and one-half tons, was crushed and screened on commercial equipment. The size grading used in preparing the aggregate is shown in Table I. Chemical analysis indicated that the aggregate contained 80% barite and 10% iron oxide. The specific gravity of the material was 4.2.

The cement used in all tests was Portland Cement A.S.T.M., Type 1.

[1] *Manuscript received May 26, 1952, and, as revised, July 21, 1952.*
Contribution from the Nova Scotia Technical College, Halifax, N.S.

TABLE I

SIZE GRADING OF CONCRETE AGGREGATE

Sieve size (Tyler)	Per cent retained			
	Series 1	Series 2	Series 3	Series 4
1½ in.	0	0	0	0
¾ in.	36	0	0	36
⅜ in.	49	29	48	49
4 mesh	66	66	65	66
8 mesh	72	72	74	72
20 mesh	83	83	83	83
48 mesh	93	93	93	93
100 mesh	98	98	98	98

Four series of concrete were tested, each series comprising three grades—low, medium, and high strength. Series 4 (see Table II) was prepared using local silica aggregate (crushed quartzite and beach sand) for comparison with the high density barite concrete. No barite was added to the aggregate in this series.

Two 6-in. diameter by 12-in. high cylinders (for compression tests) and three 6-in. cubes (for attenuation tests) were made from each batch of concrete. The cylinders were prepared according to A.S.T.M. specifications (C31-44), removed from the molds after 24 hr., stored in a moist closet for curing at 70°F. and relative humidity 90%, and then tested at 7 and 28 days in a 200,000 lb. compression machine according to A.S.T.M. standards (C39-44). The 6-in. cubes were removed from the molds after 24 hr., cured under the above conditions for 28 days, and then forwarded to the radiation laboratory.

TABLE II

COMPOSITION AND MECHANICAL PROPERTIES OF CONCRETES TESTED

Series No.	Barrier No.	Ratio of cement to aggregate		Lb. silica sand per 200 lb. aggregate	Water to cement ratio	Slump test (in.)	Compressive strength, lb. per sq. in.		Concrete density lb. per cu. ft.	Remarks
		Mix by wt.	Mix by vol.				7 days	28 days		
1	14-D	20:200	1:5.0	0	0.87:1	3	1700	2290	200	Aggregate 100% barite, maximum particle size is 1½ in.
	2-J	30:200	1:3.3	0	0.62:1	3	1955	2835	197	
	16-D	40:200	1:2.5	0	0.45:1	3	2660	3300	192	
2	13-J	20:200	1:5.2	0	0.83:1	3	1615	2425	202	Similar to Series 1 but maximum particle size is ¾ in.
	19-J	30:200	1:3.5	0	0.65:1	3.5	2190	3200	200	
	20-J	40:200	1:2.6	0	0.52:1	3.5	2975	3450	193	
3	22-D	20:200	1:5.2	44	0.75:1	3	1700	2660	184	Similar to Series 2 but local silica sand added to aggregate to increase compressive strength
	26-J	30:200	1:3.5	44	0.64:1	3.5	2165	2975	186	
	3-J	40:200	1:2.6	44	0.48:1	3	3190	4000	181	
4	21-A	20:200	1:7.6			4	1000	1670	142	Local aggregate only, no barite in mix
	7-M	30:200	1:5.0		0.66:1	3	2050	3005	145	
	27-A	40:200	1:3.8			3.5	2720	3890	145	

Attenuation of Gamma Rays in Barite Concrete

The experimental arrangement (see Fig. 1) was similar to that reported by Kennedy *et al.* (1). The radiation measuring device used was a Keleket pocket-size ionization chamber of the type commonly used for monitoring personnel in radiation laboratories. The source of radiation was a cylinder of cobalt metal. This was sealed in a machined aluminum cylinder 1.° cm. diameter and 4.6 cm. long. Its strength was 734 mgm. radium equivalent.

In order to diaphragm the radiation, a lead cylinder with a tapered hole along the axis was used. The cylinder was 18 in. long with an outside diameter of 8 in. The diameters at the two ends of the collimating hole were 3 in. and 1 in., respectively. This shield was supported in a horizontal position. The barriers were

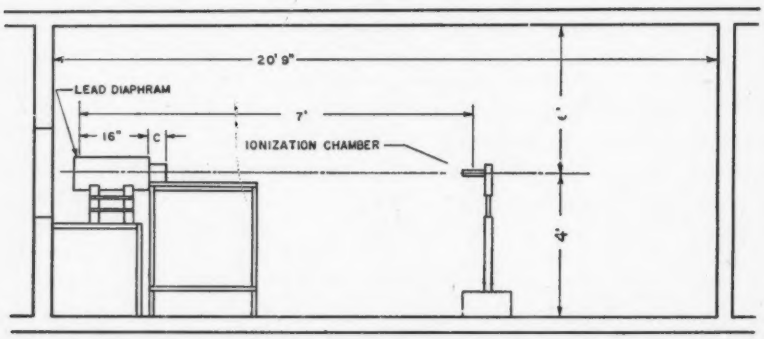

FIG. 1. Elevation of room and arrangement of apparatus for attentuation of radiation in concrete barriers. Walls, ceiling, and floor of the room, tables, and support for ionization chamber are made of wood. Barrier thickness, *C*, was varied from 3 to 18 in.

supported to fit tightly against the small end of the tapered hole. The cobalt-60 source was held at the axis of the lead cylinder by means of a wooden plug placed in the large end of the tapered hole. The position of the source was such that its center was two inches inside the lead shield.

Measurements were taken of the radiation intensity at various chamber-to-source distances (with and without a barrier in place). The inverse square relation was found to be valid for the observed intensities when the source-to-chamber distance was greater than seven feet.

Of the three 6-in. cubes prepared from each of the four concrete mixes, one block from each set was cut in half, permitting barrier thicknesses of 3, 6, 9, 12, 15, and 18 in. The radiation intensities were then measured at a source-to-chamber distance of seven feet, and the results plotted as per cent transmission versus barrier thickness on semilogarithmic paper. From the plots the half-value thickness for each barrier was obtained.

RESULTS

Working Qualities and Mechanical Properties of Barite Concrete

Table II shows the pertinent information regarding the various cylinders tested.

No difficulties were encountered in the mixing or bonding of the aggregate. The preparation and physical testing of the barite concrete, however, revealed several characteristics differing from normal experience of concrete preparation and use. These points may be summarized as follows:—

(1) In concrete made with sand and gravel aggregate failure on compression occurs along the cement–aggregate interface. The barite-concrete cylinders, however, were observed to fail across the particles of aggregate. The indication is, for barite concrete, that the maximum strength is limited by the strength of the aggregate rather than by the tenacity with which the solid particles are bonded together.

(2) The addition of a small percentage of beach sand to replace part of the fine barite increased the compressive strength but lowered the density of the resulting concrete.

(3) Cement requirements per unit volume are in direct proportion to the relative density of finished concrete. For concrete with a compressive strength of 3000 lb. per sq. in. and having a maximum particle size of 3/4 in., the proportions are:

$$\frac{\text{barite aggregate concrete}}{\text{normal concrete}} = \frac{200}{145} = 1.38.$$

(4) The work to date has been limited, but the results appear to indicate that maximum particle size of the barite should not exceed 3/4 in. if concrete having a compressive strength of from 3000 to 3500 lb. per sq. in. is desired.

Attenuation of Gamma Rays

The transmission plot shown in Fig. 2 for barrier 26-J is typical of that obtained for each concrete mix. The decreasing slope near the lower end of the graph is an indication that some scattered radiation was incident upon the ionization chamber. The amount of this radiation could have been estimated by continuing the measurements to greater barrier thicknesses until the radiation transmitted through the barrier was negligible. The apparent transmission would then be constant, independent of barrier thickness, and would correspond to the scattered radiation. However, because the experimental plot begins to decrease in slope at about 1% transmission, the amount of this scattered radiation is probably of the order of 0.5%. This is too small to produce a significant error in the half-value thickness taken from the initial part of the curve.

In Table III are recorded the half-value thicknesses for each of the 10 barriers tested. Comparison of the results for Series 1, 2, and 3 with that for Series 4 shows that a considerable reduction in the half-value thickness is obtained by using barite in place of silica aggregate. From the data reported in Table II it is apparent that the greatest reduction in the half-value thickness is observed for concrete having the highest density.

Kennedy *et al.* (1) report a mass absorption coefficient for ordinary concrete obtained using a cobalt-60 source. When the half-value thicknesses of the barriers mentioned above are calculated using this coefficient the results agree closely with the experimental values.

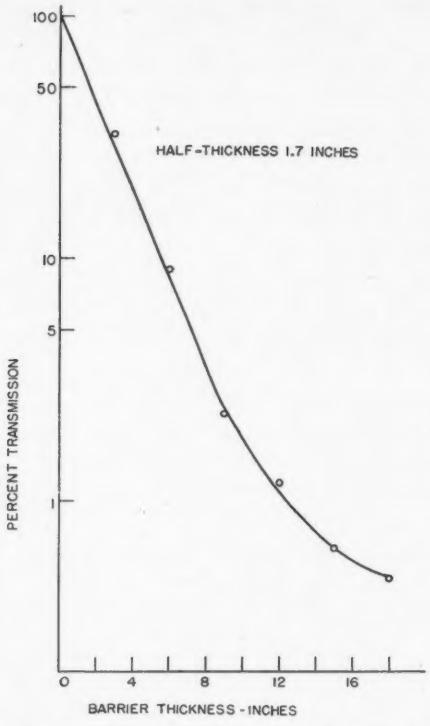

FIG. 2. Variation of per cent transmission of gamma radiation with barrier thickness.

TABLE III

OBSERVED AND CALCULATED HALF-VALUE THICKNESS FOR CONCRETES INVESTIGATED

Series No.	Barrier No.	Half-thickness (in.)	
		Observed	Calculated*
1	14-D	1.5	1.5
	2-J	1.5	1.6
	16-D	1.6	1.6
2	13-J	1.5	1.5
	19-J	1.6	1.5
	20-J	1.6	1.6
3	22-D	1.7	1.7
	26-J	1.7	1.7
	3-J	1.7	1.7
4	7-M	2.0	2.1

**Using mass absorption coefficient of 3.9 sq. in. per lb. determined by Kennedy et al. (1).*

DISCUSSION

When used as aggregate, the friable nature of barite limits the mechanical strength of the concrete. Such concrete would not be suitable for construction requiring compression strengths in excess of 3500 lb. per sq. in. Cement–aggregate bond has been observed to be good and water–cement ratio normal. Deterioration with time has not been studied.

Concrete with a density of 200 lb. per cu. ft. is readily obtained using barite aggregate. Ordinary concrete has a density of 145 to 150 lb. per cu. ft.

The narrow-beam attenuation of gamma rays from cobalt-60 in barite concrete varies directly with the density of the barrier.

ACKNOWLEDGMENTS

The investigations regarding the possible use of barite as a high density concrete aggregate were undertaken for the Defence Research Board of Canada. The authors wish to express their thanks to the members of the Board for permission to publish this paper, and particularly to Dr. A. E. Cameron, under whose general supervision the original investigation "Barite aggregate concrete in defence construction" was carried out.

It is also desired to thank J. A. Ryan and I. D. Ross, both of whom, as undergraduate students, carried out much of the detailed attenuation test work as a thesis project.

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THE DUCTILITY OF METALS IN CREEP-RUPTURE TESTS[1]

By H. H. Bleakney[2]

ABSTRACT

This paper presents evidence to show that the impurities in commercially pure metals are the most common cause of the brittle intercrystalline cracking which is characteristic of such materials in creep-rupture tests. Results of tests on copper, aluminum, and silver are given, and work on lead and iron is cited, all of which indicate decreased embrittlement, in creep-rupture tests, with increasing purity of the metal. The results on copper are regarded as being particularly significant, since they indicate that perfectly pure copper could absorb enough oxygen in the course of creep-rupture tests to become effectively impure, and thus fail by intercrystalline cracking. The "mechanical" hypothesis of embrittlement is discussed. This hypothesis attributes intercrystalline cracking to a gradual change in the mechanism of strain as the strain rate is decreased. It is argued that this hypothesis is inconsistent with the known behavior of metals.

Ductile metals, tested in tension under constant load at elevated temperatures, should, in theory, display greater total elongation before fracture the lower the applied load. In practice the ductility is usually found to be decreased as the strain rate is lowered. The decreased ductility which accompanies decreased strain rates is associated with a tendency for fissures to appear at the grain boundaries. The fissures are the obvious cause of the reduced ductility, and the purpose of this paper is to discuss the cause of the fissures.

Various hypotheses have been advanced to explain this phenomenon. Orowan (9) says intercrystalline cracking is "a consequence of the amorphous type of plasticity of the grain boundaries". Zener (14) attributes it to high stress concentration at a point where a shearing grain boundary impinges against another transversely at its end. Carpenter and Robertson (4) pointed out that recrystallization involves continuously changing grain boundaries and stated that "In the course of the adjustment cohesion at the moving boundaries is easily ruptured and intercrystalline fracture initiated". Rotherham (10) believes that, in creep-rupture tests, progressively higher strains are developed in the boundary regions which ultimately lead to the formation of fissures. It is unnecessary to extend the list of those who have written on this subject since the point in question, which they share in common, is the belief that the decrease in ductility accompanying decreased strain rates is characteristic of the metal itself, and that action by foreign agencies is not necessarily involved. These hypotheses contain the implication that the grain boundary regions behave like viscous fluids, separating grains which are regarded as behaving like rigid bodies. Bailey (1) has spoken of the mechanical nature of the grain separation, and current hypotheses are comprehended in his description.

In a previous paper the author (3) reviewed the literature dealing with creep-rupture embrittlement, and from the collected evidence concluded that the brittle

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Contribution from the Physical Metallurgy Division, Mines Branch, Department of Mines and Technical Surveys, Ottawa, Canada.
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fractures might be attributed, in some cases, to the atmosphere in which the tests were made, and in other cases to second phases concentrated at grain boundaries. In the following pages are presented and discussed the results of experiments designed to investigate the influence of both these factors.

The apparatus employed in these experiments was designed to test copper, silver, and aluminum *in vacuo* and at atmospheric pressure. The chosen metals can be broken in short times under low loads at mildly elevated temperatures; and they have the additional advantage, for experimental purposes, that they lose ductility, with decreasing strain rates, more rapidly than most other metals. The results indicated that the ductility of silver and aluminum is not appreciably affected by atmospheric gases, but it is quite susceptible to an influence which is believed to be exerted by impurities.

THE APPARATUS

Fig. 1 is a sketch of the apparatus which was designed for this work. The vacuum chamber proper is a thin-walled steel tube, flanged at both ends. The bottom end is bolted to a diffusion pump and the top to a flexible metal bellows. Both joints are sealed with neoprene gaskets. For convenience the metals are tested in the form of 16 gauge wire. The specimen is secured at each end by being wrapped around small steel rings, the distance between the wrapped ends being standardized at eight inches. The bottom ring is connected to a steel rod having a hook at the lower end so that, by manipulation of the bellows, it can be made to catch around a steel wire in the shape of an inverted V, silver brazed to diametrically opposite points on the inside wall of the vacuum tube. The top ring is connected to a brass rod which extends upward through a hole in a brass disk which closes the top of the metal bellows. The joint between the disk and rod is silver brazed. A flexible steel cable is attached to the top of the brass rod, led upward about six inches, and then horizontally across two steel pulleys which rotate on ball bearings. From the second pulley, the cable descends to a pan which holds the weights. A furnace encloses part of the vacuum tube, as shown in the illustration.

THE METHOD

Although the apparatus and the method used in these tests superficially resemble those employed in creep testing, the objective was quite different. Creep tests are made mainly in order to determine the minimum strain rate displayed by materials when extended in tension under controlled loads at elevated temperatures. In such tests temperatures, loads, and extensions must be very accurately measured. In particular the average temperature over the gauge length of the specimens should be kept within a maximum variation of $\pm$ 1°C. during the course of the test.

The present work was designed only to determine the influence of atmospheric oxygen and impurities in the metal on the ductility of copper, silver, and aluminum when strained in tension at various rates at an elevated temperature. By adopting the reduction of area as the sole criterion of ductility, problems connected with the measurement of elongation were eliminated. Load measurement

was not a problem either. Since the reduction of area decreases as the time to failure increases, each experiment involved a series of tests at different strain rates, in order that different lots of the same material, or the same material tested in different atmospheres, could be compared on a basis of equal times to failure. Therefore the absolute value of the load was not important, since the load was only the means by which the strain rate could be varied. Examination of Figs. 2, 3, 4, and 6 should clarify this point, since it is obvious that the relative positions of the lines, and not the exact position of the points, is the important feature.

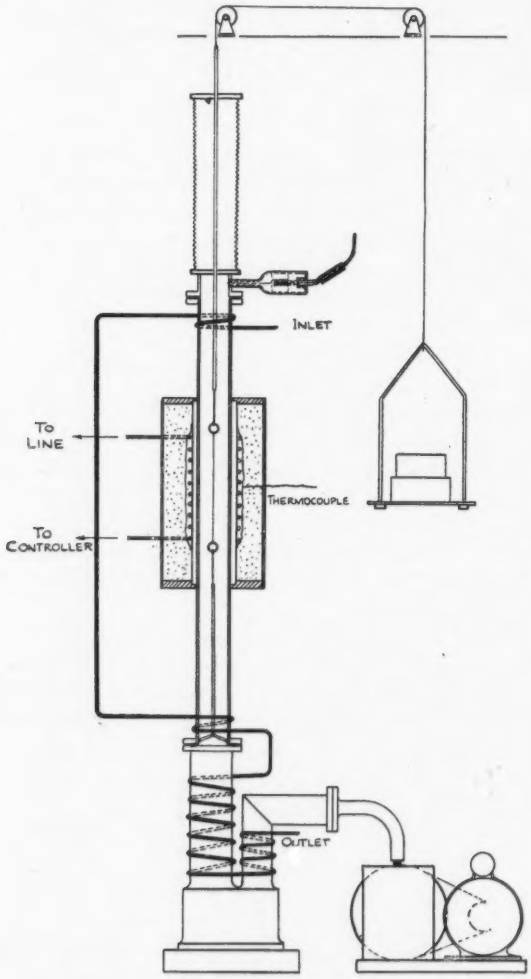


FIG. 1. Sketch of the apparatus.

Tests proved that ductility is influenced not only by strain rates but also by temperature. It was therefore necessary to provide adequate temperature control. Even here, however, a temperature difference of 8°C. did not significantly affect the results. In order to avoid the difficulties involved in trying to measure temperatures inside the vacuum tube, and also to minimize temperature fluctuations, the temperature was regulated by a sort of remote control arrangement. A thermocouple was brazed to a short cylindrical metal ring, which was clamped around the furnace, inside the loose insulation and very close to the heating element. This couple was connected to the controller. Considerable time was spent in calibrating this control couple against the actual specimen temperature inside the vacuum chamber at the hottest point. For calibration purposes only, a couple made of very fine wires was brazed to the midpoint of a standard-length test specimen which was then set up inside the vacuum chamber. The couple wires were threaded through the neoprene gasket between the vacuum tube and the metal bellows and carried to a temperature recorder. With the control point set at 425°C., the setting used in most of the tests, the following observations were made over a period of 24 hr.

Pressure, mm.Hg	Temperature, °C.		
	High	Low	Average
5×10^{-5}	352	352	352
3×10^{-2}	355	355	355
760	357	352	354

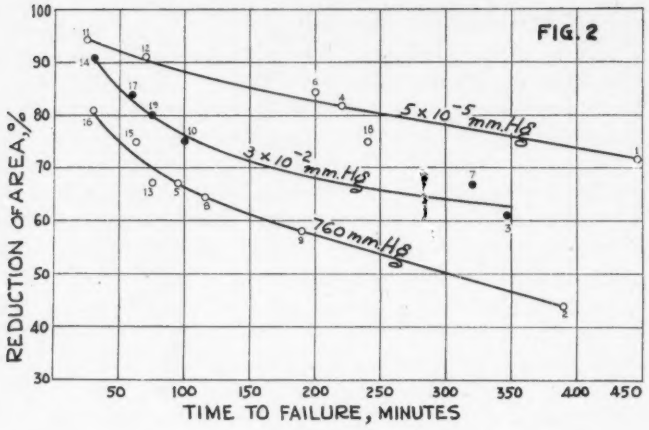
The fact that all tests were made in the same apparatus also simplified the temperature control problem.

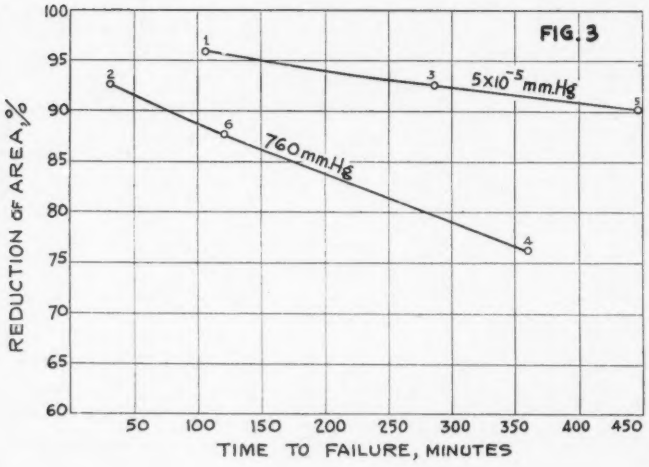
TEST RESULTS ON COPPER

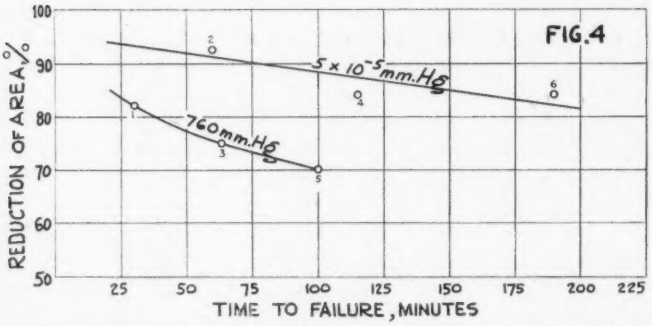
Fig. 2 illustrates the relationship which was found to exist between the atmosphere, the time to failure, and the resulting ductility of the copper used in these tests. The numbers marking the points on the curves refer to the order in which the tests were made.

Fig. 3 illustrates the results of a series of tests carried out at a temperature about 50 Centigrade degrees lower than that of the tests illustrated in Fig. 2. The increased ductility resulting from the lower temperature can be seen by comparing the curves of Fig. 3 with those of Fig. 2.

Fig. 4 illustrates the results obtained from tests carried out at a temperature about eight Centigrade degrees lower than that of the tests illustrated in Fig. 2. Under the test conditions employed, any variation in temperature between individual tests could not have been as great as 8°C.; therefore comparison of Fig. 4 with Fig. 2 will show that the differences in ductility seen in Fig. 2 at any given time to failure are attributable to the pressure of the atmosphere in which the tests were made, and not to any possible variation in temperature.







FIGS. 2-4. Creep-rupture tests on O.F.H.C. copper at various pressures with temperature control set at: Fig. 2, 425°C.; Fig. 3, 375°C.; Fig. 4, 417°C.

The curves indicate the harmful effect of the oxygen in the atmosphere on the ductility of the copper used in these tests. However, they tell nothing about the mechanism by which the ductility is impaired. Tests made on samples of less pure copper gave some evidence in this connection. Oxygen-free, high-conductivity copper was used to obtain the curves of Figs. 2, 3, and 4. Although this material was not analyzed chemically, its specific resistance was determined and found to be 1.69×10^{-6} ohm per cm.[3] Moreover examination under the microscope revealed no oxide inclusions. The second lot of material was spectrographically identical with the O.F.H.C. copper, but it contained between 0.05% and 0.10% oxygen, as estimated from microscopic examination of a polished section. The results of tests, *in vacuo*, obtained from this material are given in Table I.

TABLE I

CREEP-RUPTURE TESTS OF OXYGEN-BEARING COPPER AT APPROXIMATELY 355°C.

Time to failure	Reduction of area, %
10 sec.	83
7 min.	34
155 min.	10

The greatly inferior ductility of this material, as compared with O.F.H.C. copper, indicates that the embrittlement of the O.F.H.C. copper is not due to surface attack by atmospheric oxygen but rather to absorption of oxygen in the course of the test.

The photomicrograph shown in Fig. 6 provides additional evidence. This figure illustrates the fracture of a sample of O.F.H.C. copper tested at atmospheric pressure for three hours. Fairly general exterior cracking is evident but this cracking is superficial and has not penetrated the metal to any great extent; moreover, any stress concentration has been relieved by metal flow at the base of the cracks. The numerous internal fissures, on the other hand, increasing in size as the fractured end is approached, indicate that the final failure was of internal origin.

Table II also contributes evidence that embrittlement of copper, tested at atmospheric pressure in air, is attributable to oxygen absorbed by the metal in the course of the test. This table shows the results of creep-rupture tests on O.F.H.C. copper which had been held at the testing temperature for varying lengths of time before applying the load.

TABLE II

Holding time, hr.	Time to failure, hr.	Reduction of area, %
21	5	40
16	$4\frac{1}{2}$	45
4	6	50
1	$7\frac{1}{3}$	56

TEST RESULTS ON SILVER

Fig. 5 shows the results obtained from four different lots of silver. Preliminary tests *in vacuo* and at atmospheric pressure in air proved that the pressure of the atmosphere had little or no effect on the reduction of area. For that reason the tests illustrated in Fig. 5 were made in air at atmospheric pressure. They were made in the same apparatus as was used for copper, and with the temperature control set at 425°C. In these tests the position of the thermocouple was a little closer to the heating element than in the tests on copper. Hence the temperature of the samples was about 15°C. lower than that for the copper tests.

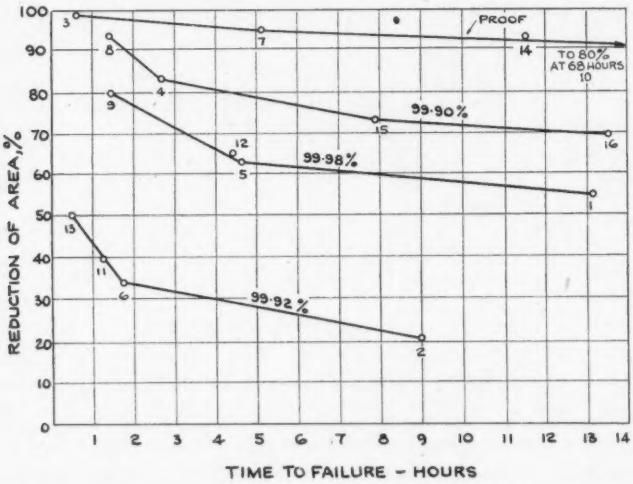


FIG. 5. Creep-rupture tests on four lots of silver. Temperature, 340°C. approximately.

Three of the four lots were commercial silver, obtained in the form of 16 gauge wire, and of the fineness shown in the figure. The fourth lot was a 2 oz. sample of proof silver obtained from the Royal Canadian Mint from material used by them as an analytical standard. Its purity was believed to be better than 99.995% although very faint traces of lead and other metals were detected spectrographically. This sample was melted by induction in an alundum crucible under lamp black and subsequently forged, rolled, and drawn to 16 gauge wire. Spectrographic analysis showed no significant difference in the purity of the silver before and after melting. All test samples were held at the testing temperature for one hour prior to application of the load. The grain size and hardness after that treatment are shown in Table III.

The most striking feature of Fig. 5 is the relatively high value for reduction of area of the highly purified "proof" silver. At the same time, the gradual impairment of ductility with time may be taken as a measure of the virulence of even extremely small concentrations of certain impurities. The lack of correlation between purity and ductility of the three commercial silvers also attracts at-

tention. The obvious and not at all surprising explanation is that some impurities are much more damaging than others. A study of the relative effects of specific impurities is not within the scope of this paper, but lies, rather, in a vastly wider

TABLE III

Fineness	A.S.T.M. grain size designation E2-39T	Hardness (D.P.H.)
Proof	.010	51.8
99.98	.015	42.3
99.92	.025	45.4
99.90	.010	49.4

field of research toward which the present investigation is intended to act only as a signpost. However a little information is available and should be reported. Table IV shows the lead, sulphur, and copper contents of the three purchased lots.

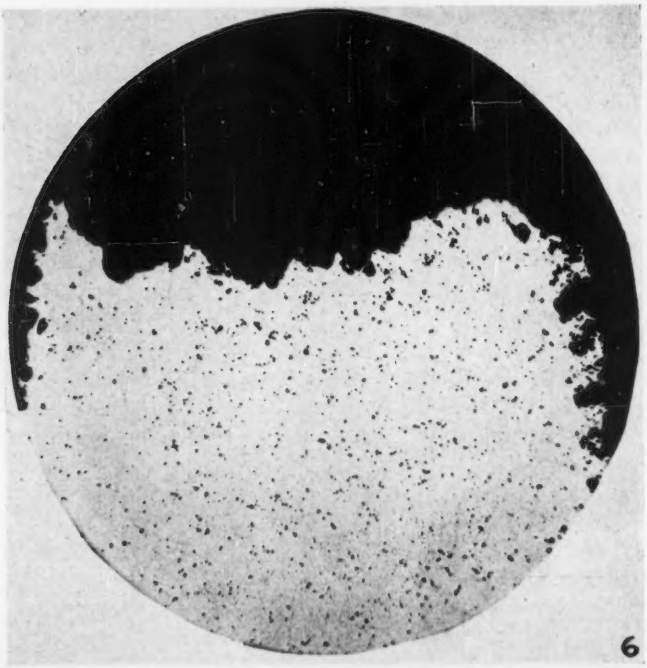
TABLE IV

% Silver	% Copper	% Lead	% Sulphur
99.98	0.006	.001	.001
99.92	0.067	.006	.001
99.90	0.100	.002	.001

The lead content of the 99.92% silver might explain its relative brittleness, but would not explain the difference between the other two lots. Possibly the high copper in the 99.90% silver is not only harmless in itself, but may even tend to neutralize the sulphur, in a manner analogous to manganese in steel. At the moment the question is highly speculative, and must await a great deal more work before the true explanation is found.

ALUMINUM

Two bars of aluminum were obtained, one being a 2S grade and the other a high purity material of 99.99 + % Al. Both bars were extruded under as nearly identical conditions as possible to a 3/4 in. round section. Small test pieces (3/16 in. in diameter and of 1 in. gauge length) were cut from the bars at a location halfway between the center and the surface. These samples were creep-rupture tested in the apparatus used for copper and silver, at a temperature of about 350°C. The examples illustrated in Fig. 7 are typical of the results obtained. The relatively brittle fracture of the 2S aluminum is seen at the left of the figure, whereas the samples at the right show that the high purity material has retained complete ductility even after much longer times to failure. That these examples are truly representative of the behavior of these materials is amply confirmed



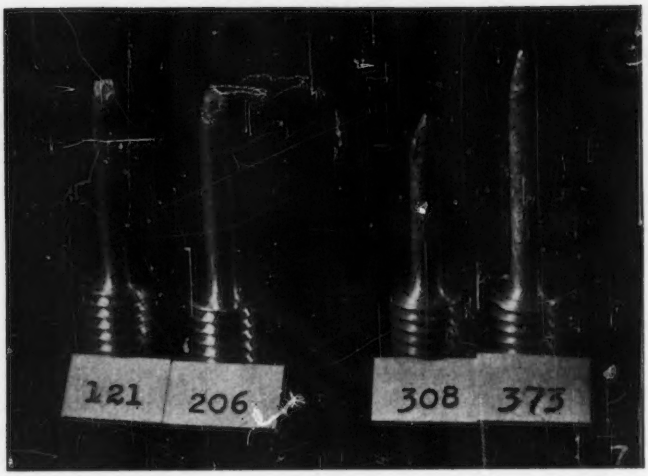


FIG. 6. O.F.H.C. copper broken in three hours at 355°C. and 760 mm. Hg. Unetched. ×75.
FIG. 7. Fractures of 99.5% and 99.99% aluminum. Figures represent time to failure in hours.

by the work of Servi and Grant (12). Although both the object of their investigation and their explanation for embrittlement differed from those of this paper, their observations are the same, and they state "(For the same reason) intercrystalline fracture is not observed in a high purity material tested under high temperature conditions".

LEAD AND IRON

Greenwood and Worner (7) have shown that a sample of 99.99 + % pure lead which failed in creep-rupture after a test lasting 439 days displayed a knife-edge fracture indicating 100% reduction of area. This fact, perhaps more than any other, prompted the author to undertake the present work, which is, in part, an attempt to answer the following question, propounded in a previous paper (3).

"Why does lead break with a fully ductile fracture after a test of 10,000 hours' duration, while copper has been found to break with a brittle intercrystalline fracture after only 81 hours or less?"

In that paper the author stated that ". . . Examination of the comprehensive work . . . (by Greenwood and his associates) forces one to the conclusion that the occurrence of intercrystalline cracking in lead is dependent upon the presence of impurities such as antimony, tin, and silver. Tellurium is especially harmful in this connection."

Jenkins and Mellor (8) have contributed evidence which indicates that iron with a relatively high sulphur–manganese ratio becomes embrittled by intercrystalline cracking in creep-rupture tests much more rapidly than iron with a relatively low sulphur–manganese ratio. It is true that the latter becomes increasingly embrittled as the time to failure increases, but the significance of the accelerating effect on the embrittlement brought about by an especially harmful impurity cannot well be ignored.

THEORETICAL CONSIDERATIONS

It is difficult to believe that the grain boundaries of a metal can lose cohesion of themselves, without the intervention of any extraneous influences other than stress and temperature. Metals solidify as crystals, and the forces which bring about solidification also constrain the atoms to arrange themselves on space lattices. Deformation distorts the lattices, but the atoms remain, nevertheless, under the influence of those forces. Thus the fact that atoms at grain boundaries are constrained to depart from their normal positions is an evidence of the operation of the forces of crystallinity, rather than a denial of them. It is, therefore, as difficult to imagine the grain boundary material behaving like a viscous fluid as it is difficult to imagine the grains behaving like rigid bodies under the stress conditions imposed in creep-rupture tests. It must be noted that when a metal, broken in tension, at any temperature, displays a reduction of area as great as 95% or more, an extension of 2000% must occur for a short distance near the fracture; and the grain boundaries must suffer this extension as well as the grains themselves. Moreover, for the same short distance, the number of grain boundary atoms must be increased at least fourfold on some of the grains. It follows that, in the course of a tension test, countless numbers of atoms, previously situated within the grains, must find themselves at grain boundaries

subjected to violent disruptive forces. Nevertheless, no loss of cohesion ensues. The idea that slower rates of strain can so alter this mechanism as to bring about intercrystalline cracking taxes the credulity.

In the "mechanical" hypothesis grain boundary weakness appears to be confused with grain boundary brittleness. Under some conditions of stress and temperature, fine-grained metals are frequently found to display greater strain rates than the same metals in the coarse-grained condition. To explain this behavior it has been postulated that at high temperatures and low stresses, the grain boundaries are weaker than the grains. This weakness has then been blamed for the intercrystalline fissuring noted in creep-rupture tests. But weakness which results in greater strain rates is very different from weakness which brings about fracture. The former is a condition of low flow-stress, the latter is an attribute of low fracture-stress. The former may be associated with the highest degree of ductility, the latter is associated with low ductility. When the cross-sectional area of a metal at the point of fracture approaches zero, as illustrated in Fig. 7, it is not difficult to equate the fracture stress with the theoretical stress necessary to overcome interatomic cohesion. Conversely it is very difficult to imagine a change in fracture strength at grain boundaries of a magnitude sufficient to account for intercrystalline fissuring. Scott, discussing a paper by Thielemann and Parker (13), made this point very cogently, and asked "How, then, can the cohesive strength of iron, or iron-base alloys, be lowered to less than 1/60 of its attainable room temperature value simply by heating to, say, 500°C.?". A simple answer to Scott's question would be that the cohesive strength could be lowered by placing at the grain boundaries a film of foreign material which itself had low cohesive strength or had poor cohesion with the metal. In any case, if the fissures were attributable to grain boundary weakness, it would have to be the weakness associated with low fracture-strength, and that is to say brittleness. But if the boundaries are brittle at elevated temperature they should be even more brittle at lower temperatures. The evidence indicates that the opposite is true.

CONCLUSION

The evidence presented in this paper points to increasing concentration of impurities at grain boundaries in the course of creep-rupture tests as the principal cause of the embrittlement noted. The behavior of copper has probably been the main obstacle in the way toward a proper evaluation of the embrittling role of impurities. Even highly purified copper displayed such a pronounced tendency to develop intercrystalline cracks that some factor other than impurities seemed to be indicated. However the results reported above show that copper can pick up enough oxygen in the course of a test to induce marked intercrystalline cracking. Schumacher (11) has recently emphasized the possible potency of even minute traces of impurities in their influence on various physical properties. Hence it is quite credible that minute quantities of oxygen can have a disproportionately damaging effect on the creep-rupture ductility of copper.

In the previous paper (3) on this subject, the author suggested that intercrystalline fracture of metals in creep-rupture tests might be due to causes similar to those responsible for stress-corrosion cracking. At the same time he

pointed out that, in some cases, the evidence pointed to grain-boundary impurities as the cause of the observed brittleness. The results reported here suggest that the latter is a more common cause than the former, and tend to confirm Greenwood's (6) suggestion that grain-boundary precipitation of impurities in lead appeared to promote intercrystalline cracking.

Before closing this paper the author wishes to speculate briefly on the mechanism of embrittlement. When impurities are the embrittling agents then the impurities must become gradually concentrated at the grain boundaries during the course of the test. This conclusion is based on the facts that the metals are highly ductile in short-time tests, and that the embrittlement is accompanied by intercrystalline fissuring which increases as time-to-failure is prolonged. Under such circumstances, either the impurities migrate to the grain boundaries, or the grain boundaries go to the impurities. The latter possibility may be worth investigating. Beck (2) and Sperry have demonstrated that in creep-rupture tests, the grain boundaries move. The question to be investigated, then, is whether or not, in the course of grain boundary migration, the boundaries collect and carry along with them some of the impurities in the metal. Such a mechanism would not be dissimilar to one proposed by Cottrell and Jaswon (5) in which they picture a slow-moving dislocation carrying along with it an atmosphere of solute atoms.

ACKNOWLEDGMENTS

The author has received a great deal of help in the course of this work, and he regrets that space will not permit adequate acknowledgment of his indebtedness to his colleagues in the Department of Mines and Technical Surveys. He must content himself with merely expressing his thanks for valuable suggestions and active assistance with the apparatus and materials, to the following members of the staff,—Dr. R. L. Cunningham, Dr. J. K. Hurwitz, Mr. H. V. Kinsey, Mr. M. J. Nolan, Mr. J. A. Fournier, Mr. J. A. Perry, and Mr. R. I. Hamilton. Grateful acknowledgment is owed to Mr. W. A. Hawkey, Chief Chemist of the Royal Canadian Mint, under whose direction the silver analyses were made. The author must also thank Dr. J. Convey, Director of the Mines Branch of the Department of Mines and Technical Surveys, for patient encouragement without which this work would not have been possible.

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NOTE

Estimation of Salt Content of Wiltshire Bacon by Electrical Resistance Measurements*

Previous work (3) has shown that the desirable salt content for export Wiltshire bacon is between 3.5 and 4.0% in the lean meat. At lower salt contents the storage life at refrigerator temperatures is reduced (2), and at higher salt levels the product tastes excessively salty. A quick method of determining the salt content of individual sides without serious mutilation would be invaluable for commercial inspection purposes. Commercial tests on the only available salinity meter designed for this purpose (1) showed that the instrument was inconvenient to use and that an increased accuracy would be desirable. An investigation of the factors affecting the correlation of electrical resistance with salt content was therefore undertaken**.

Resistances were measured on an experimental circuit consisting of an audio-oscillator, impedance bridge, amplifier, and oscilloscope. A constant voltage was applied to the bridge, and the resistance and capacitance of the circuit was balanced against that of the meat to give a null on the oscilloscope.

The resistance of bacon increased more or less exponentially with decreasing temperature, the effect of variations in temperature being marked at refrigerator temperatures but negligible at about 20°C. All samples were therefore allowed to warm to room temperature for probing.

In one test a series of seven sections of longissimus dorsi muscle containing 1.1 to 5.4% salt (by chemical analysis) were probed in duplicate with three sets of dipolar brass probes of 0.15, 0.30, and 0.60 sq. in. contact area per pole. The distance between the poles was set at one, two, three, four, or five inches, and current frequencies of 1, 10, and 50 kc. were used.

With the two larger probes, the relation of log resistance to log per cent sodium chloride was essentially linear, and the data could therefore be assessed by determining regression coefficients and residual standard deviation of the recorded readings about the calculated regression lines. With the smallest probe this relation was not apparent, so the data were not considered further. The regression coefficients, which directly reflect the sensitivity of the method, increased with decreasing pole spacing, were higher with the 0.6 sq. in. than 0.3 sq. in. poles, and were higher at 10 kc. than at 1 or 50 kc. The residual standard deviations, which inversely indicate the accuracy of the method, were smaller with the larger probe, but were not greatly affected by pole spacing or by current frequency.

* *Issued as N.R.C. No. 2848.*

** *A full account of this work is available in Report No. 52-7-1, Division of Applied Biology, National Research Laboratories, Ottawa, Canada.*

In a second trial a series of 14 pieces of muscle containing from 0.8% to 6.2% salt were probed in quadruplicate with one set (0.60 sq. in. contact area per pole) of platinum-covered brass probes. Pole spacing of one-half, one, two, three, and four inches, and a 10 kc. current were used.

The regression coefficients obtained in the second trial were considerably higher than those in the first (0.87 compared with 0.56 for comparable pole area and frequency), and were not affected by pole spacing. The residual standard deviation was least at pole spacings of one-half and one inch, and increased considerably with increasing spacings.

These data indicate that the most satisfactory conditions for determining salt content of bacon by resistance measurements are a probe size of at least 0.60 sq. in. contact area per pole, a pole spacing of one-half to one inch, and a current frequency of 10 kc. These conditions, at room temperature, gave results with an over-all error corresponding to $\pm$ 0.75% salt. In commercial practice the error would be increased by such variables as temperature (samples would necessarily be probed while cold), and differences between different lots of meat. Since exports of Wiltshire bacon have fallen to a negligible level it has not been possible to conduct tests under commercial conditions. The present work indicates that development of a resistance type salinity meter of the desirable degree of accuracy (preferably with an error of $\pm$ 0.5% salt or less) will be difficult if not impossible.

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CORRECTIONS

Page 24. In Line 3, up. $\beta = \frac{x}{2}\sqrt{Dt}$ should read $\beta = \frac{x}{2\sqrt{Dt}}$.

Page 25. In Line 2. $\frac{\beta}{x} = \frac{1}{2}\sqrt{Dt}$ should read $\beta/x = \frac{1}{2\sqrt{Dt}}$.

Page 25. In Line 5. $D = \frac{1}{4}t\left(\frac{x}{\beta}\right)^2$ should read $D = \frac{1}{4t}\left(\frac{x}{\beta}\right)^2$.

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